



Laboratory in which experimental work was conducted.

TREATMENT OF ZINC CONCENTRATE CONTAINING IRON SULFIDE.

-by-

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- A THESIS -

SUBMITTED TO  
THE FACULTY OF THE SCHOOL  
OF ENGINEERING AT THE UNI-  
VERSITY OF KANSAS IN PAR-  
TIAL FULFILLMENT OF THE  
REQUIREMENTS OF THE DEGREE  
OF BACHELOR OF SCIENCE IN  
MINING ENGINEERING.

Approved by Richard L. Grider.

May 31, 1917.



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## INTRODUCTION.

The problem of separating iron sulfide from zinc sulfide confronts the millmen and ore dressers in almost every locality in the world where the two sulfides are found occurring together in the same ore. Other valuable minerals, such as copper, lead, and silver, which may be associated with the zinc and iron sulfides, can be separated from them quite easily by the various methods of ore dressing. But zinc and iron sulfides effectively resist complete separation because of the similarity of their specific gravities. Hence other methods than water concentration must be resorted to to free the zinc blende from the iron sulfide.

The question of zinc-iron sulfide separation is found in the zinc concentrating mills of Algeria and Tunis, and in extraction plants in various places in Europe, as in Upper Silesia, Lourdes, France, etc. Ore dressers in Mexico have come in contact with this question. In the United States there are several localities which have and which are experiencing the difficulty of this separation. Wisconsin, Colorado and Missouri mills encounter this problem.

The importance of extremely pure zinc has increased enormously since the opening of the European war, and will continue to increase more than ever in the United States, since this country has entered the conflict. The specifications for munitions demand zinc, which must be well over 99 per



cent pure. Zinc ore cannot be wasted for two important reasons: (1) The enormous demand for it, and the small zinc ore reserve in the United States. (2) The high price paid for good spelter. In normal times spelter brings 4¢ to 6¢ per pound, but last year the high point for pure electrolytic spelter was 26¢ per pound, while the average price was 13¢ per pound.

The product with which we worked was table middlings of zinc and iron sulfides from the Joplin Mining district, and the object of our work was an attempt to produce a zinc concentrate free enough from detrimental impurities so that it would meet commercial requirements.

#### GENERAL DISCUSSION.

The sulfides of zinc and iron occur together quite frequently, the zinc as blende, and the iron as either marcasite or pyrite in ore bodies which are worked for the value of the contained zinc. Many lead-zinc deposits carry iron sulfide in their lower horizons. In Colorado and in the Butte district of Montana, silver and copper, as well as lead and iron sulfides are associated with the zinc blende. Gold, arsenic, antimony and bismuth compounds are also sometimes associated with the jack. All of these associated minerals except the pyrite are of economic importance, and can be separated from the zinc sulfide with comparative ease by various methods. The lead, copper, silver, gold, and part of

the bismuth can be separated by water concentration methods and flotation. The arsenic, antimony, and the remaining bismuth are volatilized, caught in flues, and baghouses and refined. As yet metallurgists have found no commercial use for the contained iron sulfides, except in some cases it is used for the generation of sulfur dioxide in the manufacture of sulfuric acid.

#### PREVIOUS WORK.

Very little information is to be had on the magnetic concentration of blende-pyrite ore or middling. We obtained the following information and references as to what work has been done and where it has been done on this problem of separating zinc-iron sulfides.

In Upper Silesia in Europe a blende-pyrite product (17 per cent zinc) is roasted and treated on two Humboldt Separators, yielding rich blende (42 per cent zinc), middlings, and a magnetic product. At Pierrefitte near Lourdes, France, and at Sakiet Sidi, Youssef, Algeria, similar plants have been erected for treating a zinc-iron sulfide concentrate. (See Eng. & Min. Jour., May 2, 1914).

In America tests on the magnetic separation of blende-pyrite ores have been conducted in 1914-1915 by the Campbell Magnetic Separation Co. of Boston, Massachusetts, in their plant at Cuba City, Wisconsin. Their reports show average

recovery (1914) of zinc to be 95.4 per cent, zinc in iron 0.6 per cent to 4 per cent, and average per cent of sulfur in iron to be 35 per cent. They claim for their process low operating costs, small amount of labor, and the adaptability to lowest grade zinc ores, table middlings, and chalcopryrite. (See booklet issued by above Company, and also Met. & Chem. Eng., Sept. 1915).

In Wisconsin and Colorado a number of companies are separating magnetic blende and pyrite with success. As far as is known, no work has been done on Joplin blende ores. However the Joplin Separating Company at Joplin, Missouri is operating a custom separating plant on blende concentrate. The iron content of raw concentrate averages 15 per cent, which is reduced to an average of 1.06 per cent in cleaned zinc product. The roasting is done in kilns, and the separation on two Cleveland-Knowles separators. (See Electro-Magnetic Ore Separation by C. Godfrey Gunther, McGraw Hill Pub. Co.).

#### SPECIAL PROBLEM.

The special problem which we have attempted to solve is the freeing the zinc sulfide from the iron sulfide. In beginning this investigation, we experimented with a middling product from mills at Alba, Mo. in the Joplin district.

Many reasons may be advanced for attempting a separation of these sulfides. The most important ones however may be classified into three general groups: (1) General or metallurgical, (2) Chemical, and (3) Commercial.



### METALLURGICAL.

(a) In the metallurgy of zinc, iron silicate is formed from the iron and silica in the charge, and attacks and destroys the retorts unless the retort is protected in some special way, as by glazing, or coating with a neutral substance, such as chromite. (b) Moreover the iron silicate is fusible, and coats more or less completely the ore particles, and thus prevents good reduction. The spelter is obtained with considerable difficulty.

### CHEMICAL.

Iron and zinc form an alloy. Iron, next to lead, is the most detrimental impurity that zinc contains. It renders spelter hard and brittle, and reduces its malleability so that it cannot be subjected to severe mechanical treatment. Very pure spelter is especially desirable at present.

### COMMERCIAL.

(a) Zinc concentrate containing more than 1 per cent iron is penalized a certain amount per ton by the smelter. (b) Impure spelter brings a much lower price than the better grades.

Many difficulties are experienced in treating a problem of this nature. Very little information suggesting methods of attack is obtainable. The problem requires a great deal

of time in order to work out nicety of details. Lack of information of the small amount of previous work, brevity of time and inadequacy of equipment have handicapped us in this work.

We proposed to solve this question by roasting the material used, and then subjecting the roasted product to magnetic concentration, or preferential flotation separation.

The magnetic compounds of iron formed by roasting the sulfide are strongly magnetic and are attracted by fields of low intensity, but as the quality of separation made depends entirely upon the uniform magnetic quality of the material presented to the magnet, the roasting is the most important step in the process.

Upon roasting marcasite with access of air, a portion of the sulphur is driven off as sulfur dioxide ( $\text{SO}_2$ ) and the non-magnetic  $\text{FeS}_2$  is transformed, superficially at least, to the form  $\text{Fe}_7\text{S}_8$  (analogous to pyrrhotite), which is strongly magnetic. Care must be taken with the roast because it is very easy to obtain a roasted product of uneven permeability. One way to overcome uneven permeability in the resulting material is to roast a sized product.

If the sulphur is completely driven off by the roast, the oxide  $\text{Fe}_3\text{O}_4$  results, which is strongly magnetic. Should the roast be carried farther, the oxide  $\text{Fe}_2\text{O}_3$  results, which is quite feebly magnetic. These two oxides of iron pass from one to the other, according as the atmosphere of

the furnace is reducing or oxidizing.

Marcasite begins to lose sulphur and change over into the magnetic sulfide, and finally over into the oxides at temperature of about  $370^{\circ}\text{C}$ . The roast must be conducted below  $600^{\circ}\text{C}$ ., the ignition point of blende. Should  $600^{\circ}$  temperature be exceeded, the only result is a superficial oxidation of the blende. Should a temperature of say  $620^{\circ}\text{C}$ . be maintained for some time, however, a serious loss would result through the oxidation of the fine particles of blende.

A prerequisite of success in any separation process is the existence of the minerals to be separated as free particles and in this magnetic separation constitutes no exception.

The concentrate should be sized before roasting. Roasting and magnetization take place from the surface inward, and in a mass of ore composed of coarse and fine particles, the finer sizes will have been over-roasted before the lumps have been effected to their centers. In the case of a lump ore with a medium roast (98 per cent of product used in these tests passed a 35 mesh screen) the larger lumps will have centers of unchanged marcasite, while the fine particles may have been converted into the non-magnetic sesquioxide, and it is evident that a clean separation of such a product is out of the question. With very fine material, the interstitial spaces are small, and the material has



a tendency to pack, hence to permit the gases good contact with the particles; the ore must be well rabbled and stirred. C. Godfrey Gunther states that certain experimenters have given 8 mesh as the best size for blende marcasite concentrate for good results in roasting. Hence decided difficulties were experienced with the concentrate we used since as above stated, most of it was 150 and 200 mesh material.

#### SOLUTION OF PROBLEM.

The ore used in these tests was an Alba, Missouri table middlings which results from passing the crushed zinc ore over the concentrating tables at least two times. The ore is the typical rosin jack associated with a dark green marcasite and a small amount of flint chert and limestone gangue. It occurs in the sheet ground of this district. Blende is isometric-tetrahedral in crystallization. The marcasite crystallizes in the orthorhombic system.

The jack has a vitreous lustre and ranges in color from a light yellow to a dark brown. The larger particles are the darker in color. It occurs in angular particles and flaky grains. The marcasite is dark green in color and very much more finely divided than the blende. The gangue is mostly in fairly large splintery particles which can be thrown out almost completely by screening the material through an 80 mesh sieve. Specific gravity of blende after roasting was 4.291 and of the roasted marcasite 4.035. Assays show the ore to contain 24.63 per cent zinc and 30.45 per cent iron. The lime

and insoluble runs between 1 and 2 per cent. (By microscope).

#### METHOD OF ATTACK.

##### Roasting With Magnetic Separation.

##### General Discussion and Theory of:-

Marcasite or pyrite  $\text{FeS}_2$  (marcasite and pyrite do not differ except in their systems of crystallizations and very slightly in the specific gravities) does not possess sufficient permeability to be attracted by even the most intense magnetic fields. There are two methods for rendering iron sulfide magnetic: (1) A slight roast with the formation of the magnetic sulfide or (2) a more complete roast with the formation of a magnetic oxide of iron.





Screen Analysis of Original Sample  
Cut Out By Jones Riffle Sampler (Fig.1).

Total Wt. = 9807 Gram.

Product size mesh.	Weight in grams.	per cent of total	pounds per Ton	Tons per 1000 tons.
+10	6	.06	1.2	.6
+35	196	2.00	40.0	20.0
+80	945	9.63	192.6	96.3
+150	2136	21.78	435.6	217.8
+200	2295	23.40	468.0	234.0
+200	<u>4209</u> = 9787	<u>42.92</u> 99.79	<u>859.4</u> 1996.8	<u>429.2</u> 997.9

Loss in Grams = 20 Grams

Per Cent Loss = .21 per cent

The product on 10 mesh was negligible and consisted principally of gangue.

The 35 mesh product contained considerable gangue but little iron. The iron was finer than the Sphalerite.

The 80 mesh product was practically the same as the 35 mesh product.

The product on 150 mesh contained little iron and had a very high Sphalerite content. Color was yellow with dark specks of iron scattered throughout.

The 200 mesh product contained a great deal of iron and was a dark yellowish color.

The product through 200 mesh contained very little zinc and was nearly pure iron sulfide.

# Assay of Screen Products Before Roasting.

Product	Zn per cent	Fe per cent
All thru 80	22.49	30.18
All on 80	23.50	24.64
On 80 mesh	22.42	26.52
On 35 mesh	28.77	15.65
On 150 mesh	41.28	17.27
On 200 mesh	28.39	26.89
Thru 200 mesh	10.16	40.61

## Residue Analyses After Roasting. (Best Cleaned Product).

Product	Zn per cent	Fe per cent	Time of Roast
On 80	29.80	7.40	5 min.
On 35	34.17	6.64	5 min.
On 150	62.76	1.60	5 min.
On 200	63.62	4.31	15 min.
Thru 200	44.20	15.36	6 min.
All on 80	32.47	7.03	5 min.
All thru 80	60.99	7.46	7.5 min.

The mode of attack of the problem by magnetic separation was begun as is consistent with good roasting practice. Realizing that the sizing of the material was necessary, a screen analysis was made using the 10, 35, 48, 80, 150 and 200 mesh screens. It was found that 88.1 per cent passed through the 80 mesh screen and 42.92 per cent passed through the 200 mesh screen. The concentration of the Sphalerite in the different products was very noticeable. The greatest concentration was

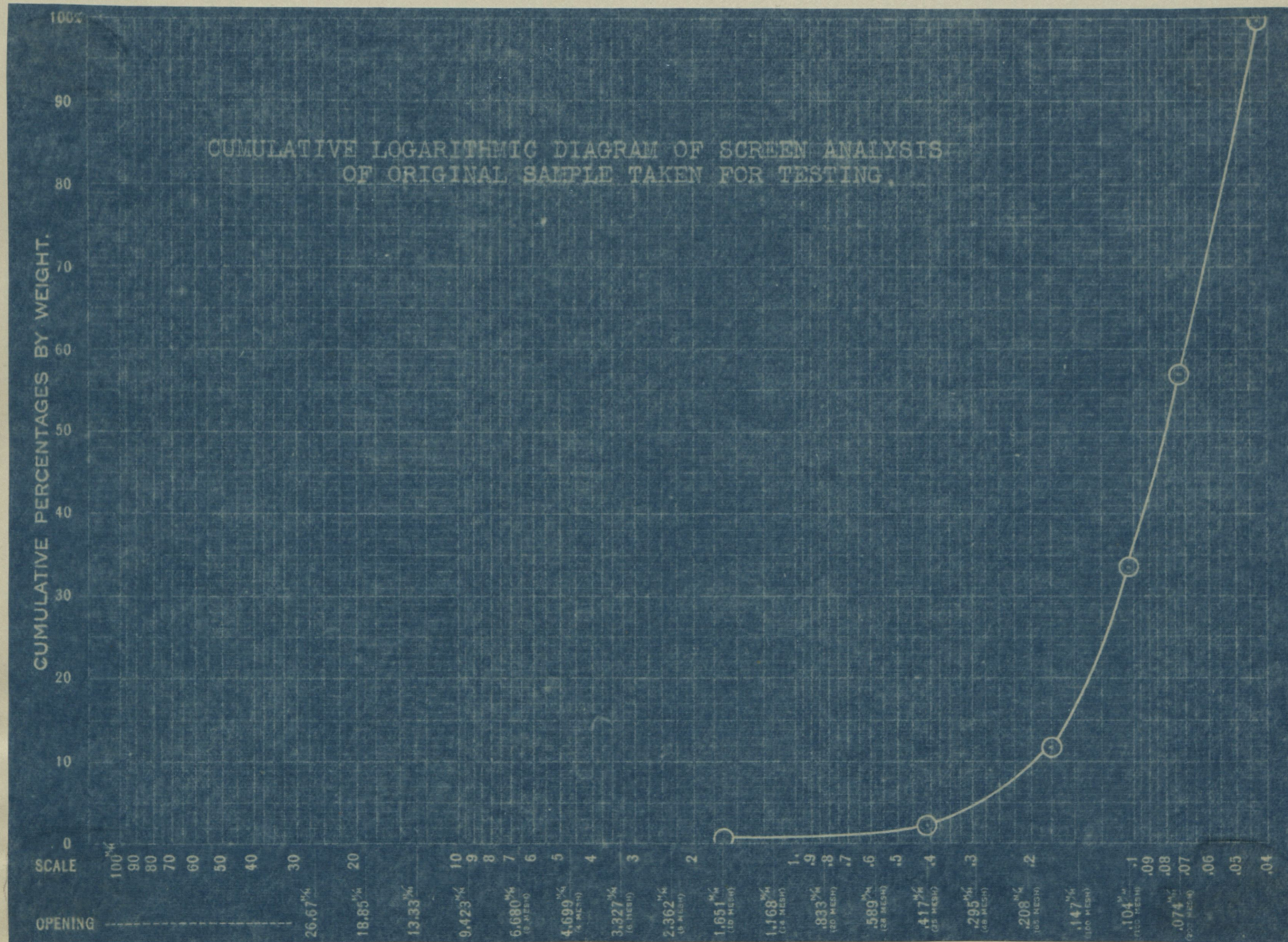


Fig. 1.

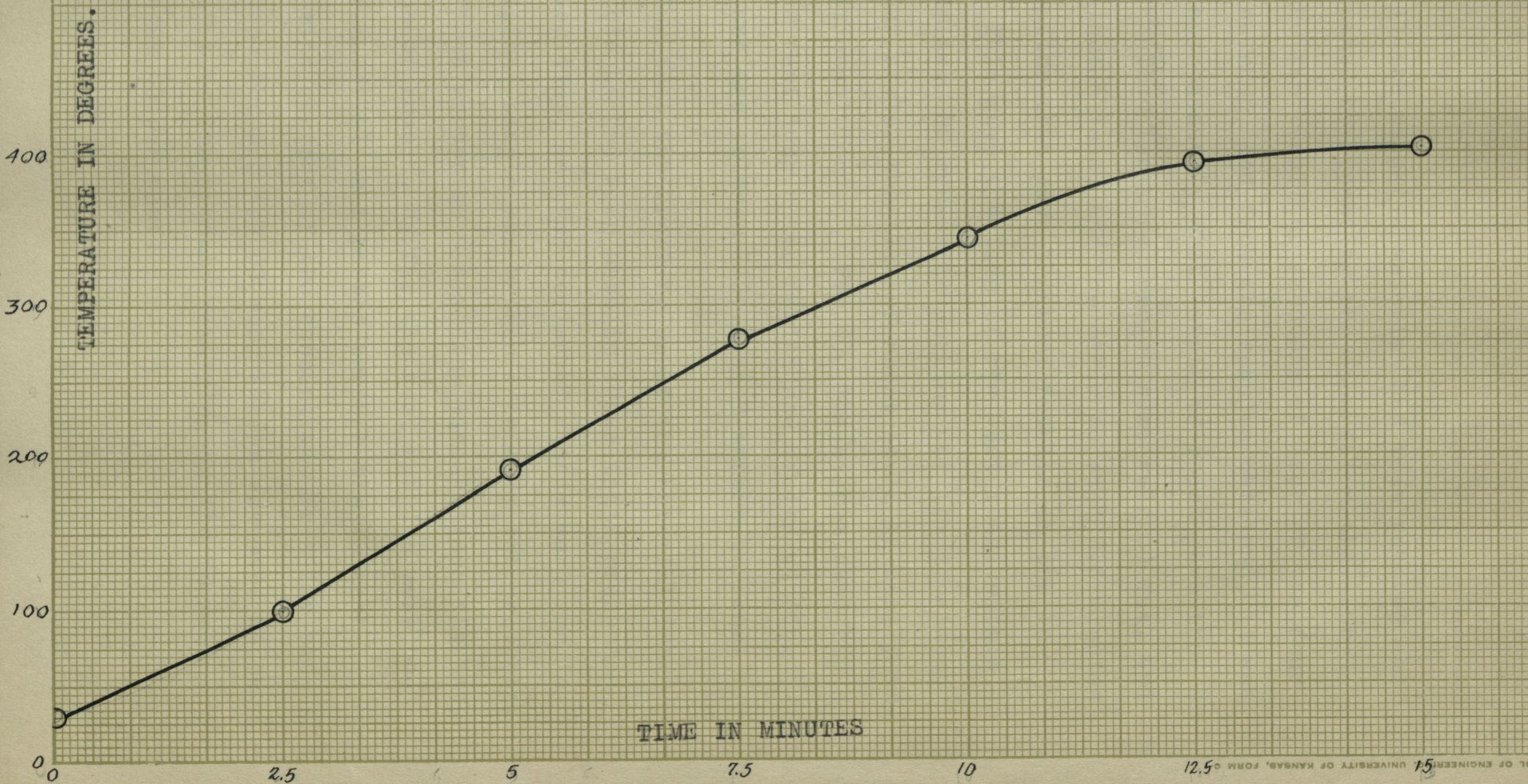
in the material on 150 mesh which assayed 41 per cent zinc, while the minimum was reached in the product through 200 mesh which assayed only 10 per cent zinc. Another noticeable feature was the nearly complete absence of gangue in the product that passed through the 80 mesh screen. Considerable limestone and chert was separated out on the 35 mesh screen.

After running a screen analysis on the ore, the actual work of magnetic separation was started. In order to determine if any of the material could be separated magnetically without previous roasting, a magnet was passed through the different products, but the only material that adhered was very fine iron or steel dust that had probably come from the rolls or the jaw crushers used in breaking the ore.

At first it was thought best to do all roasting in a muffle furnace but a little investigation showed that the problem of temperature control would be exceedingly difficult, so that plan was abandoned. In muffle furnaces much trouble is experienced in obtaining an excess of air to give a true oxidizing roast. The use of roasting dishes and a Bunsen burner seemed to be the next best method and was found to work very well. Four samples of 10 grams each of the different screen sizes were weighed out and various time lengths of roast given to each of the four samples. The length of roasts were generally 5, 10, 15 and 20 minutes. Each of these samples was then treated with a large electromagnet, and all magnetic material separated out.



CURVE SHOWING TEMPERATURES DURING THE GRADUAL  
ROAST OF A SAMPLE.





After all the magnetic material had been removed and thoroughly cleaned by repeating the separation three or four times, the two resulting products of each of the four samples were weighed, and from the results obtained, it was easy to find approximately what length of roast gave the best results. Then two more samples were weighed out, and one roasted 2.5 minutes less, and the other roasted 2.5 minutes more than the time that gave the best results in the initial roast. This gave the time of the best roast within very close limits. Of course in working out the treatment on a commercial scale, the time of roast could be easily adjusted to meet conditions. (Fig.2) The magnet used had a 1 inch core 2 feet long. The resistance of the winding was .57 ohms and the voltage 110, with 2 ampere current.

Realizing that the breaking up of the material into as many products as had been used in these tests presented serious difficulties, another series of roasts were run in which the original sample was broken into only two products, using the 80 mesh screen as a dividing line between the two. In this series of roasts, by far the best results were obtained in the material that passed through the 80 mesh screen. The zinc content of the concentrate from the product that passed through the 80 mesh screen was over 64 per cent, while that of the product remaining on the 80 mesh screen was about 33 per cent.

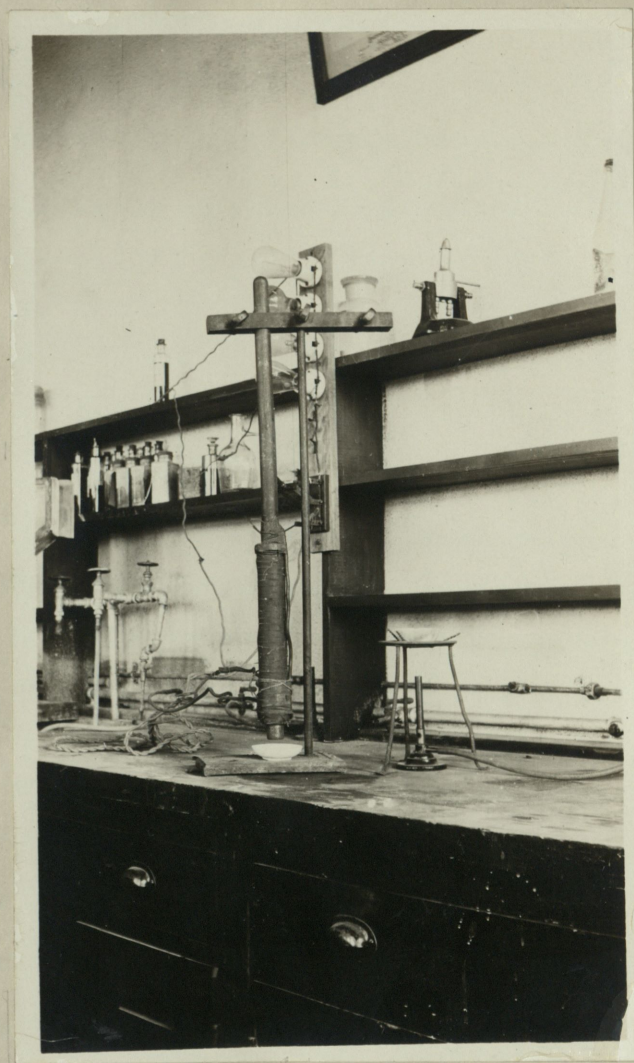


Fig. 2



Gradual Roast of Product on 35 Mesh.

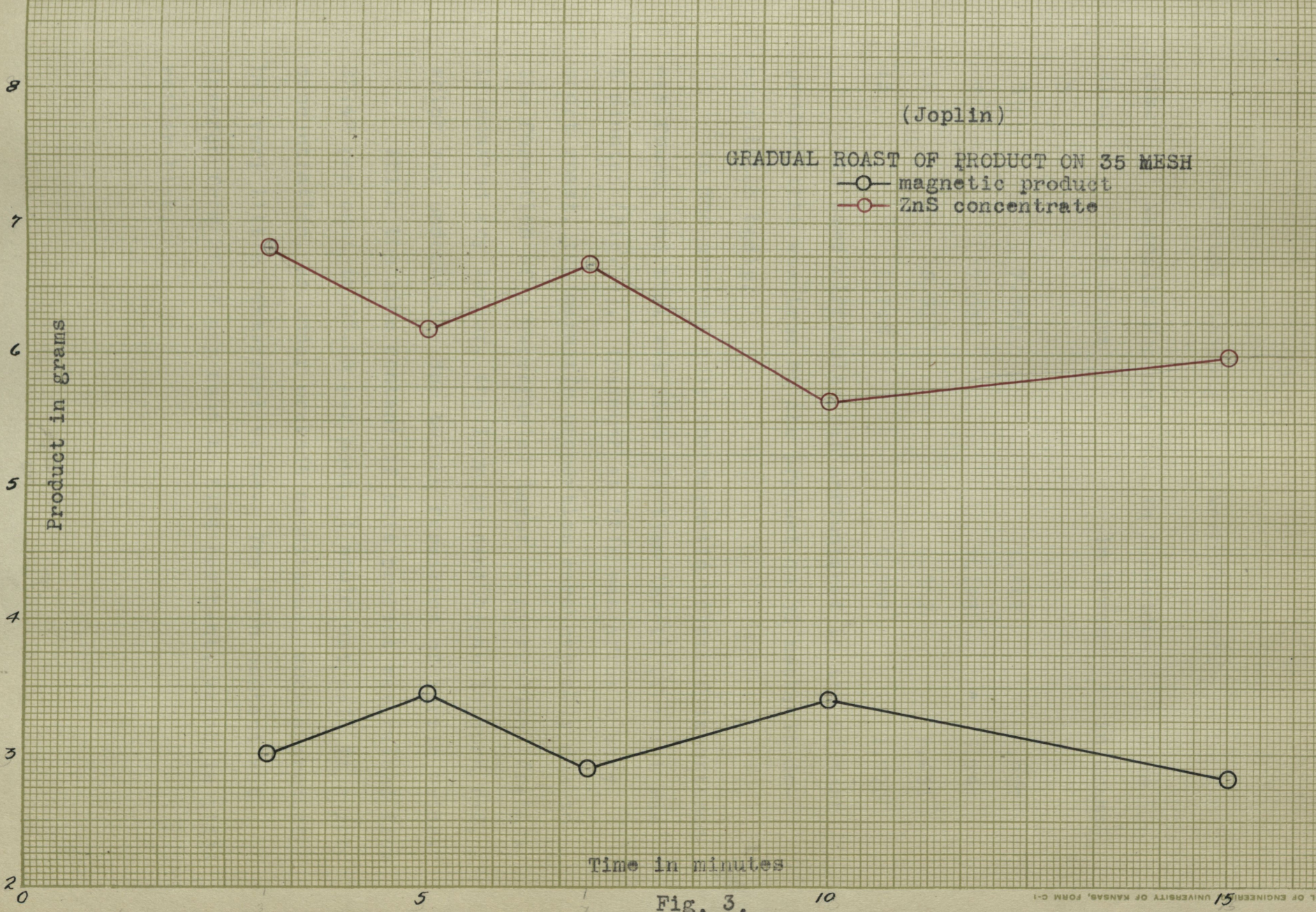
Time	Magnetic Product in Grams	Zn S Residue in Grams	Loss in Grams
3	2.991	6.814	.195
5	3.458	6.182	.360
7	2.892	6.681	.427
10	3.413	5.649	.938
15	2.821	5.997	1.182

In the roasting of the 35 mesh product, considerable difficulty was experienced through the decrepitating of the gangue in the product.

By far the best time length of roast was 10 minutes. The curve of the roast (Fig.3) shows two points, five and ten minutes, at which the extraction was good. The good separation at 5 minutes was probably due to the formation of superficial pyrrhotite ( $\text{Fe}_7\text{S}_8$ ) or magnetic sulfide of iron. The point of the other good extraction was probably due to the further oxidation of the pyrrhotite to the magnetic oxide of iron overlying a layer of unaltered pyrrhotite. When the roast was carried over 10 minutes, the iron present changed from a black to a reddish color, and at the same time became less magnetic. This was probably due to the formation of haematite or ferric oxide ( $\text{Fe}_2\text{O}_3$ ).

The rise in concentration of the zinc in this product was small, being from 28.77 per cent to 34.17 per cent. The iron was reduced from 15.65 per cent to 6.64 per cent, but is quite high, considering the low zinc content.





(Joplin)

GRADUAL ROAST OF PRODUCT ON 35 MESH

—○— magnetic product  
—○— ZnS concentrate

Time in minutes

Fig. 3.



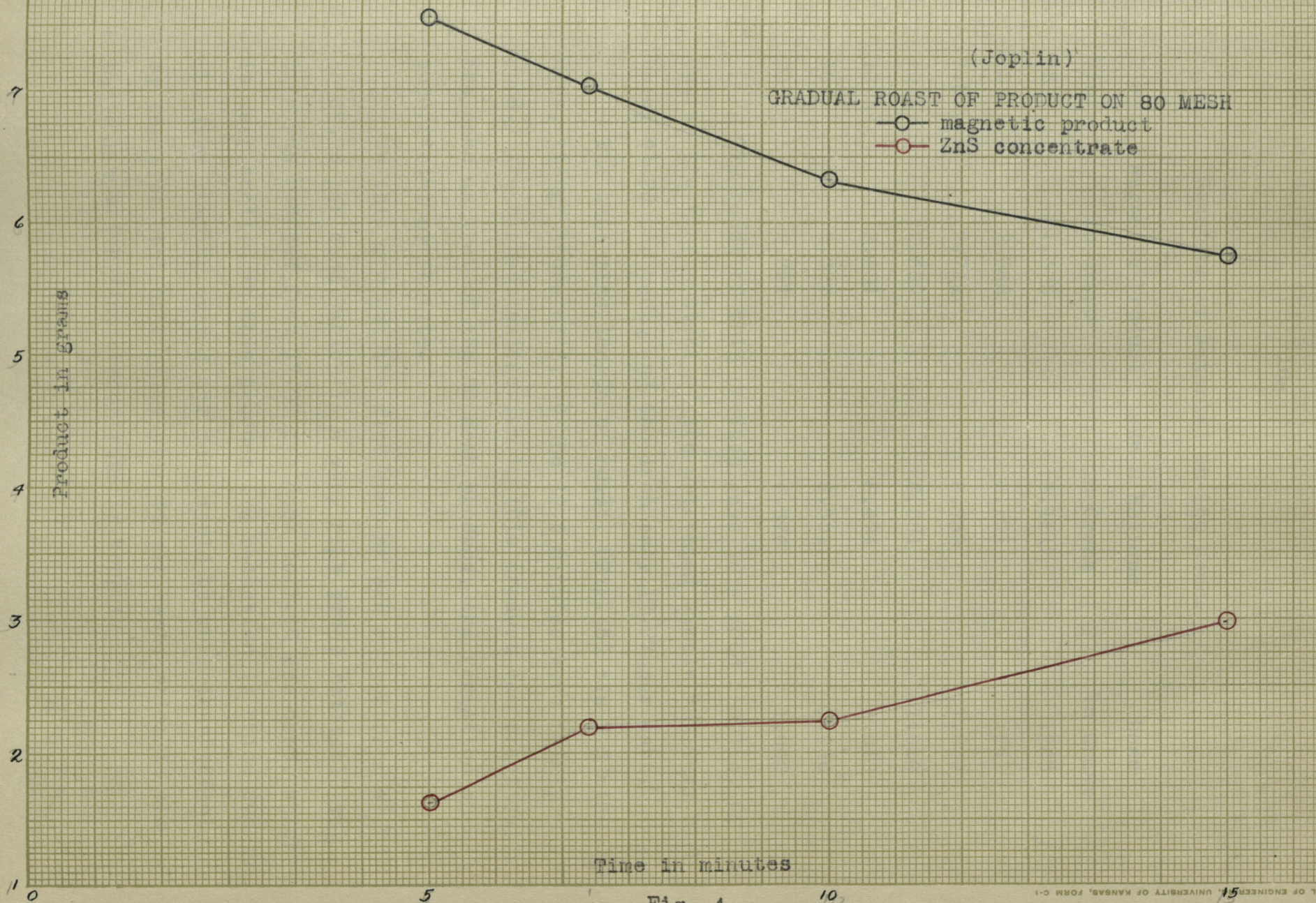
Gradual Roasting on 80 Mesh Product.

Time	Magnetic Product	Zn S Residue	Loss
Min.	in Grams	in Grams	in Grams
3			
5	7.551	1.642	.807
7	7.032	2.183	.785
10	6.317	2.232	1.451
15	5.743	2.987	1.270

The same difficulty was encountered in the 80 mesh product that was true of the product on 35 mesh; it decrepitated badly.

In the product only one magnetic point of importance was found, and that was when the time length of roast was 5 minutes (Fig. 4). Inasmuch as this was after a very short roast, the magnetic properties were probably due to the formation of a superficial layer of pyrrhotite ( $\text{Fe}_7\text{S}_8$ ). If roasted any longer, the iron present began to turn red, which was undoubtedly due to the formation of ferric oxide.

While the zinc content of this product was raised from 22.42 per cent to 29.80 per cent, I would not call it a good concentration, as under present conditions, such a product has very little commercial value. The iron content was lowered from 26.52 per cent to 6.64 per cent, which is still rather high.



Time in minutes

Fig. 4.



Roasting of Material on 150 Mesh.

Time	Magnetic Product	Zn S Residue	Loss
Min.	in Grams	in Grams	in Grams
3	1.575	8.161	.263
5	2.968	6.391	.641
7.5	2.711	6.250	1.039
10	2.609	6.381	1.010
12.5	2.484	6.474	1.042
15	2.383	6.581	1.036
20	2.391	6.576	1.042

The roast of this product gave very good results. Of course the zinc content of the untreated sample was very high being 41.28 per cent, and the iron only 17.27 per cent.

The best time length of roast, (Fig. 5) was 7.5 minutes, though those of 5, 10 and 12.5 minutes were fairly good. The highest extraction in this product was practically the same as that of the 80 mesh product, the formation of superficial pyrrhotite.

In this product the zinc content was raised from 41.28 to 62.76 per cent, which is a very high grade material. The iron was reduced from 17.27 per cent to 1.60 per cent, which is very small. In this product the reduction in price due to the iron running over 1 per cent would be more than offset by the zinc content running over 60 per cent, which is the basis upon which zinc concentrate is bought.



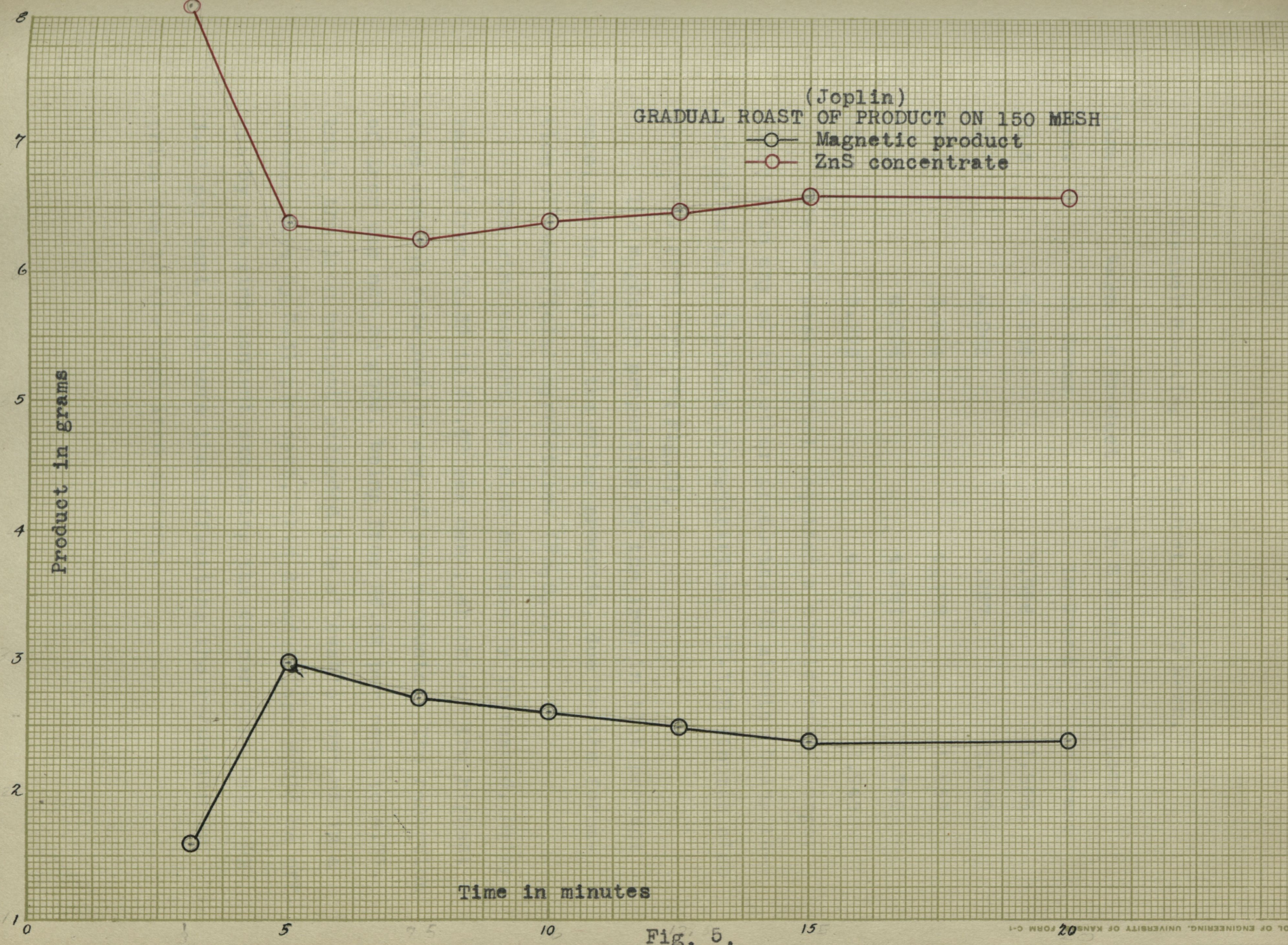


Fig. 5.



Gradual Roasting of Product on 200 Mesh.

Time	Magnetic Product	Zn S Residue	Loss
Min.	in Grams	in Grams	in Grams
12.5	4.136	3.996	1.868
15	4.263	3.957	1.780
17.5	3.814	4.328	1.858
20	4.004	4.231	1.765
25	3.976	4.079	1.945

The 200 mesh product required a considerable longer roast than any of the other products; 15 minutes being required to give the best concentration. 12-1/2 minutes also gave very good results (Fig.6).

The magnetic properties of the iron in this product were, as in most of the other products, due to the formation of superficial pyrrhotite. ( $\text{Fe}_7\text{S}_8$ ).

The zinc content was also materially increased, being raised from 28.39 to 63.62 per cent, which was the highest zinc content obtained in any of the products. The reduction of iron was not so good, being from 26.89 per cent to 4.31. While the iron content exceeded the allowable content in a concentrate by 3.31 per cent, this disadvantage would be offset by the zinc content exceeding the 60 per cent basis by 3.62 per cent, and more than offsetting the reduction in price due to the high iron content.

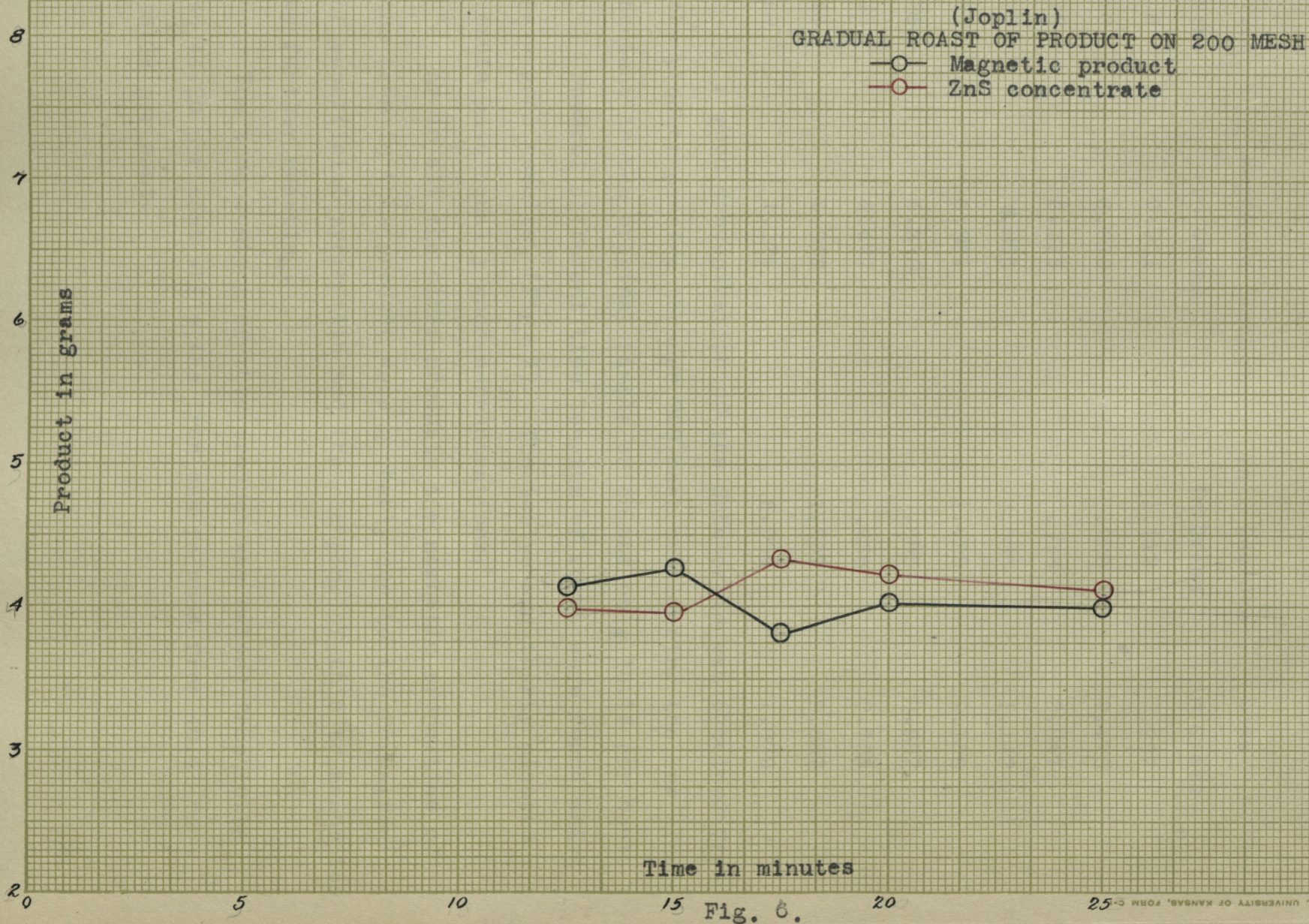


Fig. 6.



Gradual Roasting of Product Thru 200 Mesh.

Time	Magnetic Product	Zn S Residue	Loss
Min.	in Grams	in Grams	in Grams
6	7.901	1.112	.987
10	6.610	.920	2.470
12	6.315	.838	2.847
15	5.805	3.083	1.112
20	6.391	1.423	2.186
25	6.310	.853	2.837
30	6.427	.838	2.735
45	5.926	1.207	2.867
60	1.176	6.954	1.870

A good concentration of the Sphalerite was obtained from the product passing through 200 mesh considering the low zinc content in the original sample which was only 10.16 per cent.

Several different time lengths of roast were found which gave good results. (Fig. 7). The twelve and thirty minute roasts gave equally good concentrations.

As in all the other products the magnetic properties of the iron in this product were probably due to the formation of superficial pyrrhotite and the changing of this to magnetite by further roasting.

The zinc content of this product was raised from 10.16 to 44.20 per cent while the iron was reduced from 40.61 to 15.36 per cent.





Gradual Roast of Products on 80 Mesh.

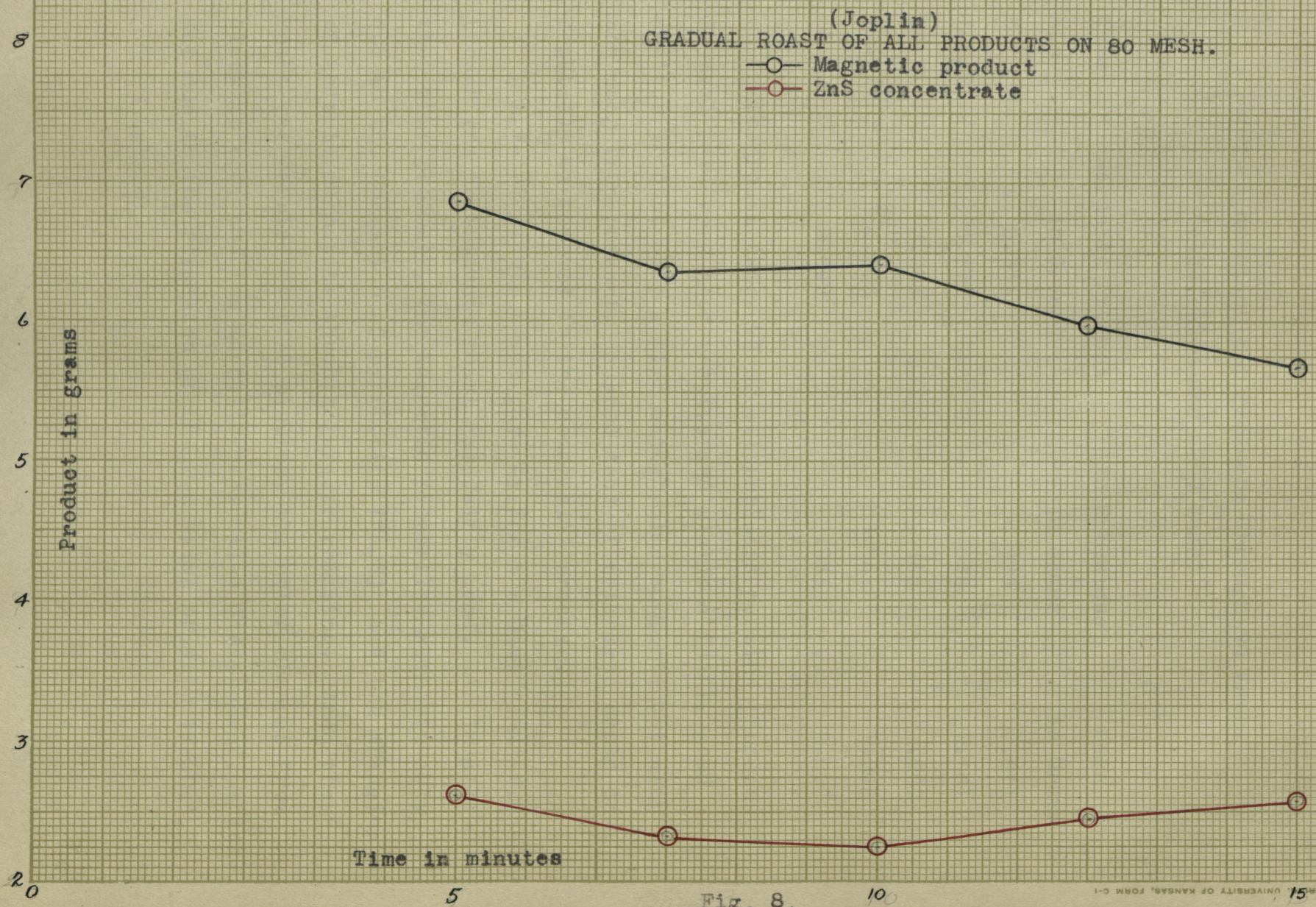
(+35 and +80 mesh)

Time	Magnetic Product	Zn S Residue	Loss
Min.	in Grams	in Grams	in Grams
5	6.871	2.664	.465
7.5	6.365	2.331	1.304
10	6.435	2.248	1.317
12.5	5.975	2.452	1.573
15	5.737	2.591	1.672

The variety of all products on 80 mesh gave rather poor results (Fig.8) as the resulting product was not very high in zinc and contained a fairly large per cent of iron.

The best time length of roast was found to be 10 minutes. The magnetic properties of the iron sulfide were undoubtedly due to the formation of superficial pyrrhotite. When the roast was run over 12 minutes the iron began to turn from black to red, due to the formation of the red ferric oxide.

The zinc content was raised from 22.49 to 32.47 per cent, while the iron content was reduced from 24.64 to 7.03 per cent.



Time in minutes

Fig. 8.



Gradual Roast of all Products Through 80 Mesh.

(+150, +200 and -200 Mesh)

Time	Magnetic Product	Zn S Residue	Loss
Min.	in Grams	in Grams	in Grams
5	5.331	4.058	.611
7-1/2	5.771	3.068	1.161
10	3.935	5.565	.500
12-1/2	4.303	3.251	1.946
15	5.042	2.951	2.007

The roast (Fig. 9) of all products passing through 80 mesh gave very good results and yields a fairly good smelter product with a high enough zinc content, but with too much iron present. A good deal of this iron could undoubtedly be removed in the commercial process.

A 7-1/2 minute roast gave by far the best results, though the 12.5 minute roast was fairly good. As in all the other products, the magnetic properties were probably due to the formation of pyrrhotite ( $\text{Fe}_7\text{S}_8$ ) and the changing of this to magnetite through further roasting. When the roast was carried over 15 minutes, the color changed from black to red, and the magnetic properties disappeared, which was due to the formation of the red oxide of iron ( $\text{Fe}_2\text{O}_3$ ).



8  
7  
6  
5  
4  
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2

(Joplin)  
ALL PRODUCTS THRU 80 MESH

—○— Magnetic product  
—○— ZnS concentrate

Product in grams

Time in minutes

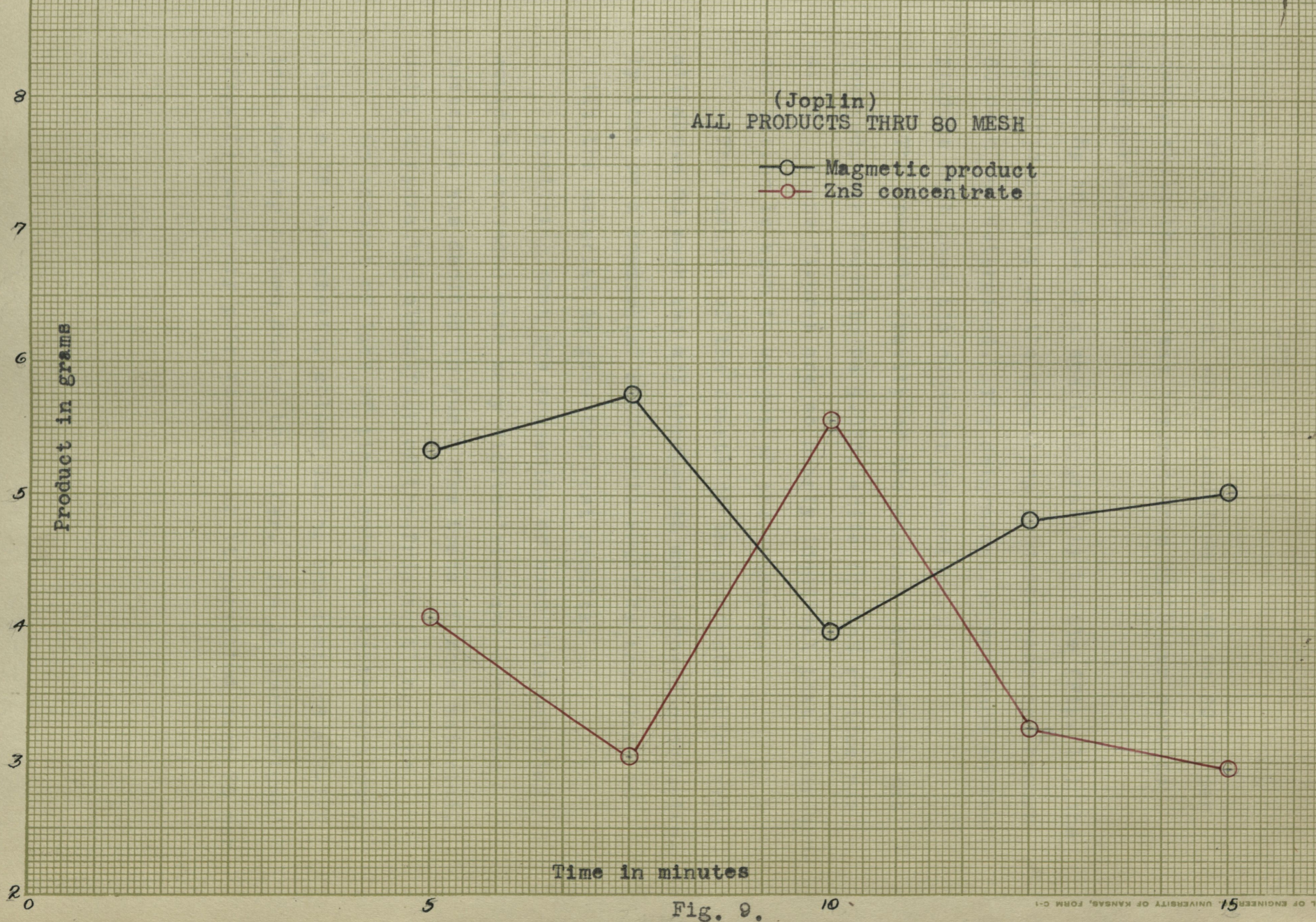
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Fig. 9.

10

15

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The zinc content was raised from 22.49 to 60.99 per cent, while the iron was lowered from 30.18 per cent to 7.46 per cent.

By the use of a specific gravity bottle (Fig. 10) the specific gravity of the resulting products passing through 80 mesh was determined. The determination was made on the product that gave the cleanest concentrate.

The specific gravity of the magnetic iron which was removed, was found to be 4.035, while the ZnS residue was 4.291. These figures are so nearly identical that water concentration is seen to be out of the question.

The results of the roasts of the products which were all above 80 mesh and all through 80 mesh showed conclusively that on a commercial scale this would be the place to split the material to give the best results.

Realizing that the screening of such fine material would be attended with many difficulties and excessive costs, it was decided to run a few tests of this material, using a hydraulic classifier of the free settling type.

On account of lack of time, only two tests were run, using a 16 inch sorting column during one test and a 9-1/2 inch sorting column during the other. The 16 inch column (Fig. 11) gave very poor results, as it permitted over 25 per





Fig.10



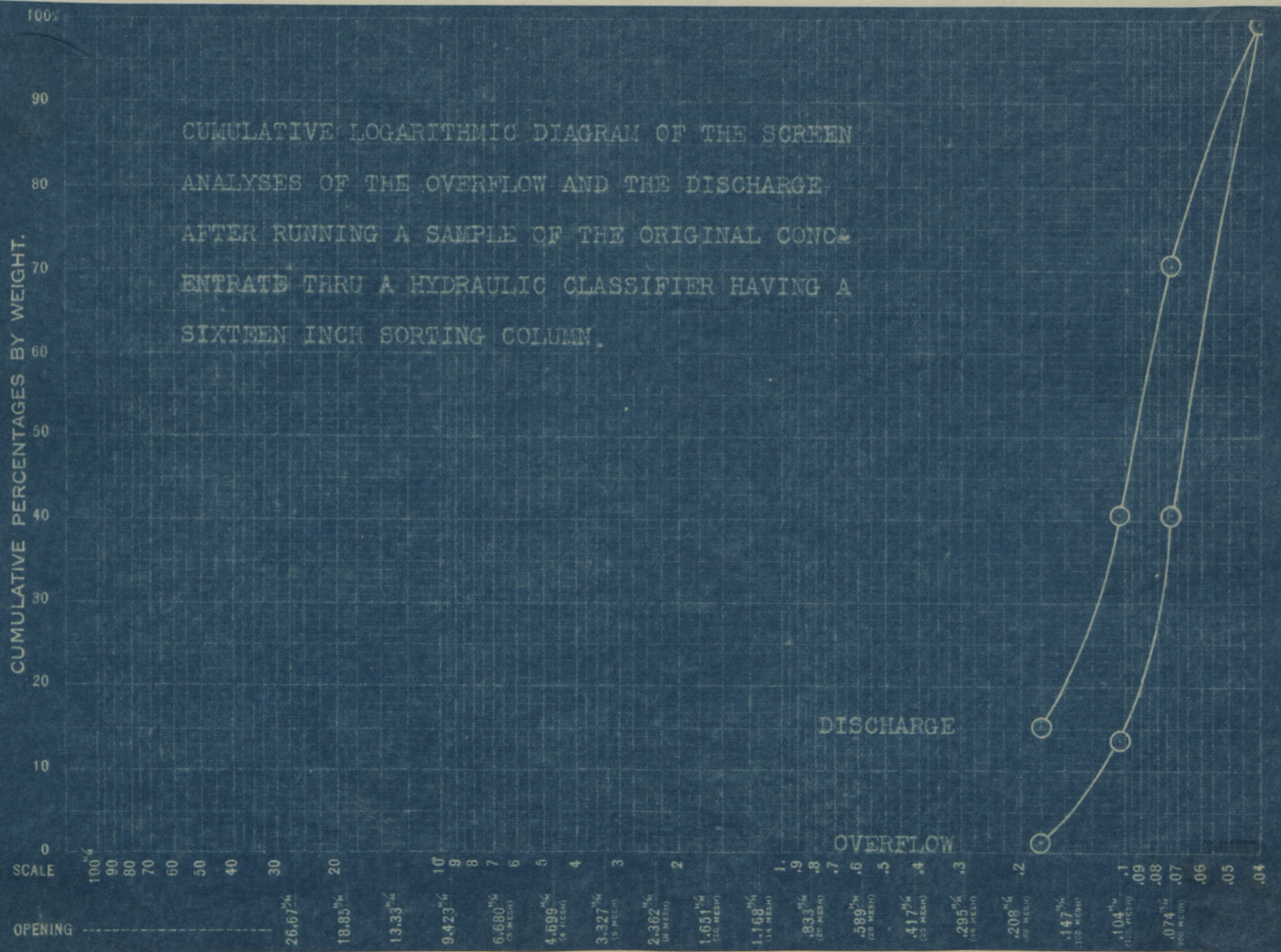


Fig. 11.



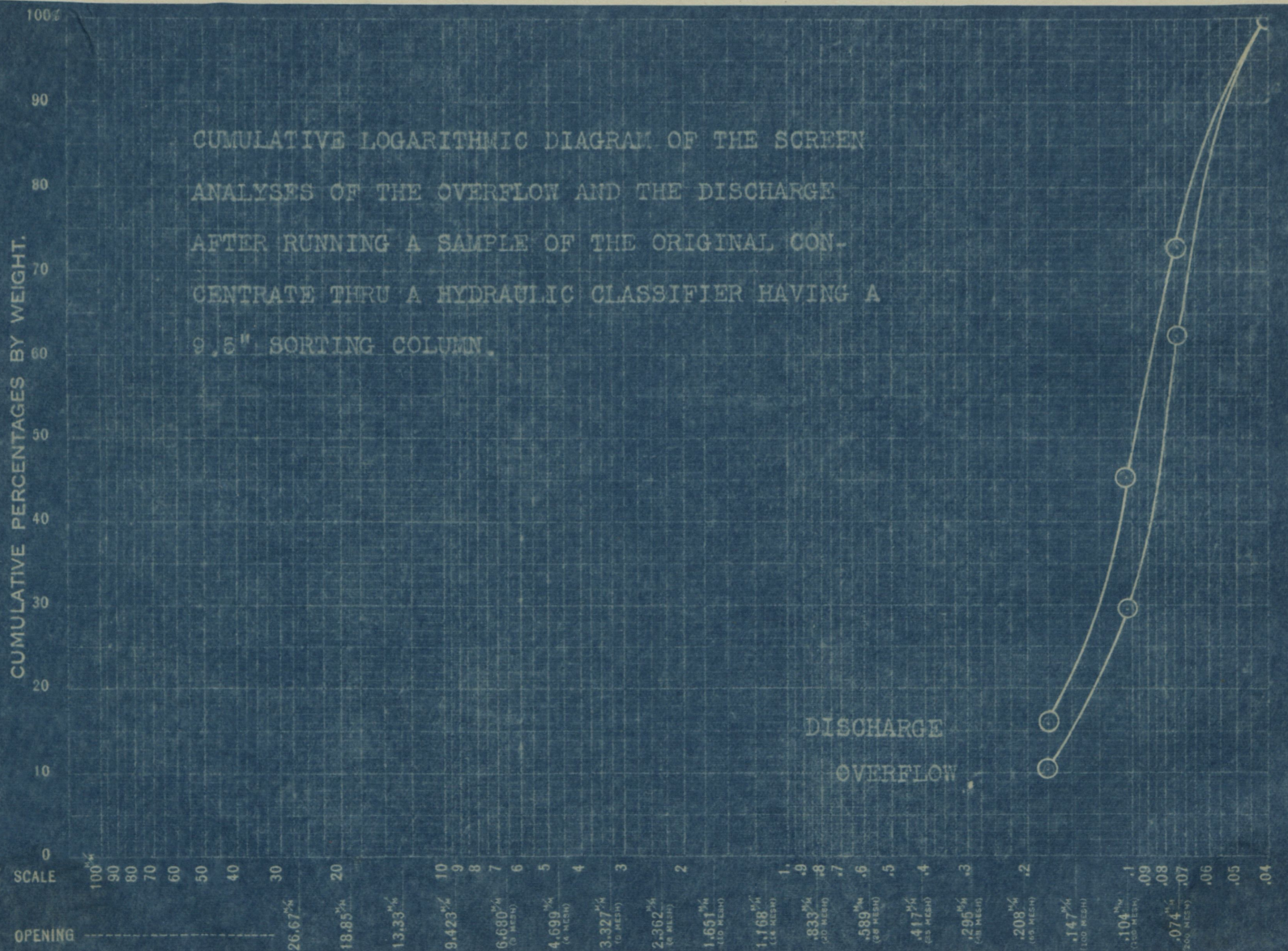


Fig. 11a.

cent of the product through 200 mesh to go to discharge. The 9-1/2 inch column gave a fairly good classification, as the accompanying table will show.

The classifier used (Fig. 12) was a hydraulic free settling classifier with a peripheral water feed, which gives the water in the sorting column a rotary motion. The classifier, as the accompanying illustration shows, was a laboratory size, and is capable of giving very accurate results on small samples.

#### WATER CLASSIFICATION.

(16 inch Settling Column)

#### OVERFLOW.

Product	Weight in Grams	Per cent	lbs. per Ton	Tons per M.Tons
on 80	3	1.43	28.6	14.3
on 150	26	12.44	248.8	124.4
on 200	56	26.79	535.8	267.9
Through 200	<u>122</u>	<u>58.37</u>	<u>1167.4</u>	583.7
	207	99.09	1980.6	

Loss = 2 Grams or .97 per cent





Fig. 12

DISCHARGE.

Product	Wt.	per cent	lbs. per Ton	Tons per M. Tons
On 80	92	15.10	302.0	151.0
On 150	156	25.61	512.2	256.1
On 200	182	29.87	597.4	298.7
Thru 200	<u>179</u>	<u>29.39</u>	<u>587.8</u>	<u>293.9</u>
	609	99.97	1999.4	999.7

WATER CLASSIFICATION.

(9-1/2 inch Sorting Column).

(OVERFLOW)

Product	Wt.	per cent	lbs. per Ton	Tons per M. Tons
On 80	51	10.40	208.0	104.0
On 150	94	19.18	383.6	191.8
on 200	159	32.45	649.0	324.5
Thru 200	<u>184</u>	<u>37.55</u>	<u>751.0</u>	<u>375.5</u>
	488	99.53	1991.6	995.8

Loss = 2 Grams or .42 per cent

(DISCHARGE)

Product	Wt	per cent	lbs. per Ton	Tons per M. Tons
On 80	44	15.77	315.4	157.7
On 150	68	24.37	487.4	243.7
On 200	80	28.67	573.4	286.7
Thru 200	<u>85</u>	<u>30.46</u>	<u>609.2</u>	<u>304.6</u>
	277	99.27	1985.4	992.7

Loss = 2 Grams or .73 per cent

From the results of the different tests it is easy to see that the best concentration obtained on the undersized product after the original sample had been split on the 80 mesh screen.

The problem is a large one, and of course in the short time allotted, it was impossible to go as much into the details as should have been done. The main thing that has been accomplished has been the demonstration that the Joplin zinc and iron sulfide concentrate can be freed of iron by magnetic separation. Another important point that was clearly brought out was the necessity of classification before roasting. This was most clearly shown by the different time lengths of roast that each product required, but which seemed to take a large jump at the 80 mesh screen.

The strength of magnet used also impaired the results to a certain extent on account of its being too strong and to a certain extent attracting the Zn S. In further investigation along this line a magnet of variable strength over a wide range would be advisable.

The main thing to be worked out in solving the problem would undoubtedly be to take up the question of classification:

#### FLOTATION DISCUSSION.

It is not yet five years since the starting of the first American mill using the frothing method of flotation, yet



55,000 tons of ore is being treated daily by this process in the United States to-day. However not much work has been done upon the preferential flotation of roasted zinc-sulfide ores. Preferential flotation is a specialized application of the flotative principle in the separation of minerals from their ores. Selective flotation means the flotation of valuable minerals in presence of undesirable gangue minerals. Preferential flotation is the flotation of one of the ordinary selectively flotative minerals in presence of another similar mineral.

Sulfides are the only mineral compounds it has been found possible to float. In many instances an ore is roasted to oxidize one of its metallic sulfides to the oxide, and then it is subjected to flotative separation in which the unaltered sulfide minerals are floated while the oxide of the other mineral remains behind in the tailings.

In the tests we performed, we proposed to roast the iron sulfide to the red ferric oxide form ( $\text{Fe}_2\text{O}_3$ ) and floating the unaltered zinc sulfide while the iron would come off as tails.

#### PROCEDURE.

About 2000 grams of the ore were screened out to pass an 80 mesh sieve. This was roasted on a sheet of asbestos over gas burners. The assay of the roasted product showed 27.89 per cent zinc and 29.90 per cent iron.

A number of test-tube tests were made upon the ore to determine the amount of oil and acid to use. Oil and acid were used together.

A typical test is as follows:-

Weigh out 10 Grams of ore (through 80 mesh)

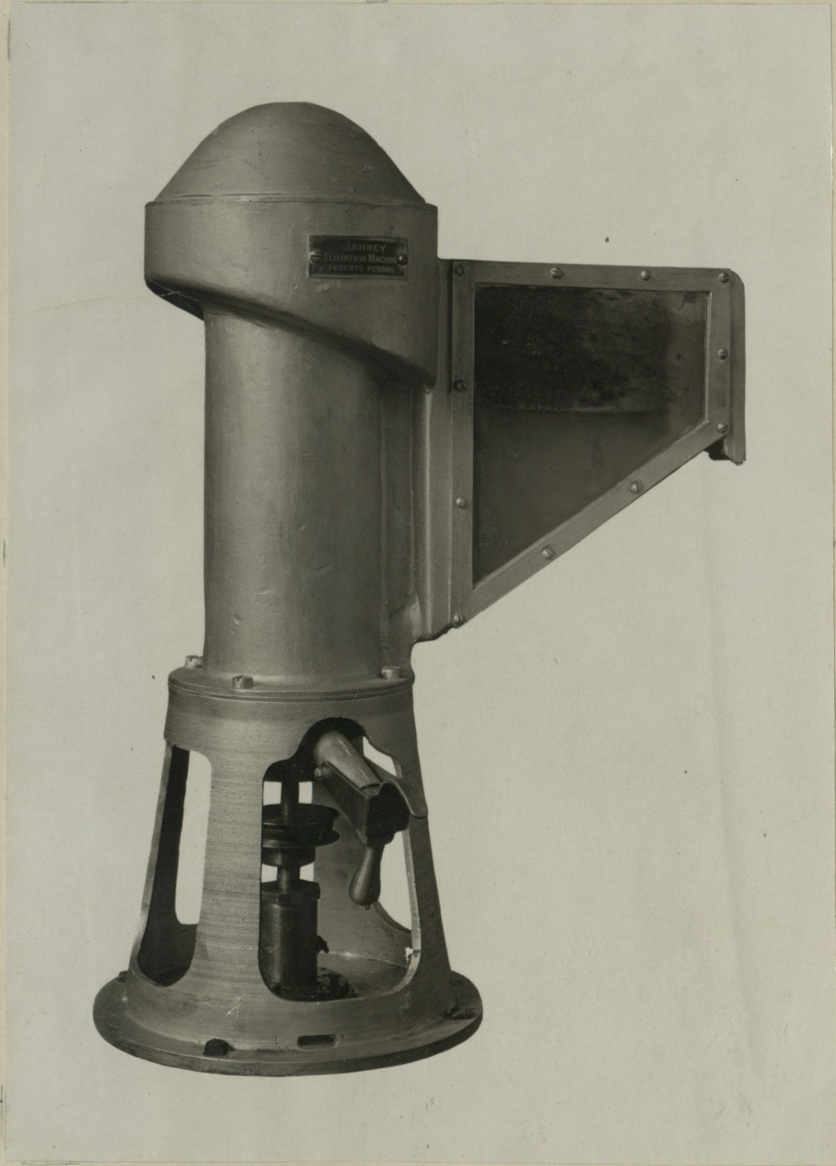
measure out ( 30 c.c. water  
(  
( .1 c.c.. sulfuric acid from burette  
(  
( .01 c.c. pure oil from dropper (70 drops=1 c.c.)

Shake above mixture energetically in a 100 c. c. test tube or graduate cylinder for a quarter to a half minute, and note results.

After a satisfactory treatment had been determined upon by test tube and bottle experiments, the following work was done with a Janney flotation machine in the laboratory. (See cut).

#### PROCEDURE WITH MACHINE.

Start motor and set rheostat for lowest speed. Run enough clear water in machine to just show in spitz. Now add 500 grams of the sample. The cover on the machine is placed in the inverted position. This is done to allow thorough mixing without circulating the pulp. All of the required amounts of oil and acid are now added, and the motor brought up to full speed. Mix in this way for about 30 seconds. Bring motor back to low speed again and invert cover so that round side is up. The machine is again brought up to full speed and water added through hole in cover until the created froth reaches overflow lip in spitz. The froth is skimmed off with small porcelain slate. Sides of glass in spitz are wet down as the froth is rising, so that it will not stick to the glass.



Janney flotation machine used in  
flotation tests.

The test duration continues for about five minutes, during which time the mineral bearing froth is skimmed off as created. The concentrate froth may be caught in a pan, placed under the lip.

The machine is now brought back to slow speed and the tailing plug removed. The tailing is caught in a pan as it runs out the opening.

#### JANNEY MACHINE--SPEEDS OF MOTOR.

	Slow Speed	Full Speed
No Water in machine	1400 r.p.m.	1800 r.p.m.
With water	1024 r.p.m.	1668 r.p.m.
With water and ore	900 r.p.m.	1500 r.p.m.

#### RESULTS.

Acid and Oil were used together. Hydrochloric acid gave better results with pine oil than sulfuric acid, probably because the former generated more gas with iron oxide than the latter. One c.c. of HCl was used per 100 grams of ore. Eight drops of pine oil No. 265 per 100 grams of ore brought best results.

Flotation concentrate assayed on average 64.20 per cent zinc and 1.44 per cent iron. Between 85 per cent and 90 per cent extractions of the zinc sulfide were obtained in the small tests. The above results were checked quite closely in the tests in the Janney machine.



## FLOTATION RESULTS.

The results of the flotation tests showed conclusively that the possibilities of solving the problem by this method are good.

The labor and cost of roasting the material preparatory to floating is small and the flotation itself is very economical.

Inasmuch as the time allowed would not permit the working out of the details of the method such as the kind and amount of oils, acids and reagents to be used, further investigation along this line should take in the solution of these details.

## COMPARISON OF RESULTS.

Flotation in the tests conducted gave better results than magnetic separation. Both gave concentrates with the zinc content over the desired 60 per cent, which is the basis upon which zinc concentrate is bought, but the iron was not reduced within the allowable one per cent by either method, though the flotation method brought it nearest to the desired per cent.

Exact comparison of the results of the two methods may be made from the following figures which are the best for the two processes.

Roasting and Magnetic Separation of all  
through 80 mesh.

Zn = 60.99 per cent

Fe = 7.46 per cent

Roasting and Floating all through 80 mesh.

Zn = 64.20 per cent

Fe = 1.44 per cent

#### RECOMMENDATIONS.

We suggest very strongly that further work should be done upon this problem of zinc-iron sulfide separation by either magnetic concentration or by flotative separation.

Suggested subjects for research in roasting and magnetic concentration.

- I. Suitable roasters and temperature regulations.
- II. Different strengths of magnet.
- III. Permeability of metals.
- IV. Suitable separators.

#### FLOTATION RESEARCH.

- I. Proper Oils
- II. A number of laboratory tests with different machines in order to obtain comparisons.

GENERAL.

- I. Tests from Ores amenable to these treatments from different districts in order to determine the treating of the different classes of ore efficiently
- II. Afterwards an extended investigation of the methods, costs, and relative efficiencies of the various processes should be worked out for the purpose of determining the most economical method both from the physical and financial points of view.